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Tensile properties of nanosilica/epoxy nanocomposites

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Abstract

The effect of nanosilica on the tensile stress-strain response of Epikote 828 epoxy polymer was studied. A 40 wt% nanosilica/epoxy masterbatch was used to prepare a series of nanocomposites with 5–25 wt% nanosilica content. Static uniaxial tensile tests were conducted to investigate the tensile stress–strain response and tensile properties of unmodified and nanomodified epoxy polymers. In addition, the degree of dispersion of the silica nanosphere particle in the epoxy matrix was investigated using transmission electron microscopy. It was found that the incorporation of a well-disperse nanosilica improved the tensile properties of the polymer. The addition of 25 wt% nanosilica enhanced the tensile modulus and strength of about 38% and 24%, respectively, compared to the neat polymer without sacrificing the failure strain.

Keywords: Nanocomposites; nanosilica; epoxy resin; stress-strain response; tensile properties.

1. Introduction

The latest development in polymer technology is nanocomposites whereby the addition of nano-sized fillers into the polymer, such as epoxy, can lead to a number of desirable effects. The most practical reasons [1-6] are to:

- stiffen the matrix (increase elastic modulus) and make it more rigid,
- enhance strength and failure strain,
- improve resistance to crack initiation and propagation (fracture toughness), and
- reduce the coefficient of thermal expansion, thermal shrinkage and enhance thermal stability of the neat polymer that could reduce thermal stresses and hence fibre waviness of fibre reinforced composite laminates.

In order to achieve these, the selected nanofillers usually have higher elastic modulus and lower coefficient of thermal expansion than the matrix. There are several types of nanofillers commercially available and commonly used for developing nanomodified-epoxy, such as montmorillonite organoclay, nanosilica, carbon nanotubes (CNT) and carbon nanofibres (CNF). Silica nanospheres are used as a reinforcing filler for polymers because of their high elastic modulus (70GPa), high specific surface areas (50-380 m^2/g), high thermal stability (1200^{o}C), low density (1.8 g/cm^3), low thermal expansion coefficient, good abrasion resistance and low material cost (USD $8.50/lb) [1,3,7-9].

Epoxies are widely used as the matrix in fibre reinforced polymer (FRP) composite. In theory, a stiffer and tougher epoxy matrix provides a good load transfer and lateral support to the fibres, gives better resistance to fibre instability (microbuckling) and delays crack initiation and propagation [10-11]. Therefore, by modifying the resin using nanofillers will improve the overall matrix-dominated properties such as in-plane shear, interlaminar shear and compressive strengths

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and fracture toughness. However, many factors affect the capability and performance of the nanomodified-resin. It primarily depends on the type and properties of the fillers along with their surface treatment, compatibility of the fillers to the epoxy and hardener, degree of dispersion of the fillers in the matrix and their interfacial adhesion, processing methods and curing conditions [1-6,9-12]. Improper selection of resin, fillers and fabrication method leads to a significant reduction in the overall properties of the composite laminates; nanofillers may act as manufacturing defects.

In this study, the effect of pre-treated nanosilica on tensile stress-strain response and tensile properties of the Epikote 828 epoxy system was investigated. The degree of dispersion of the nanofiller in the matrix was evaluated. Additionally, the influence of nanoparticle dispersion on the tensile properties was also discussed.

2. Experimental details

2.1. Fabrication of nanosilica/epoxy nanocomposites

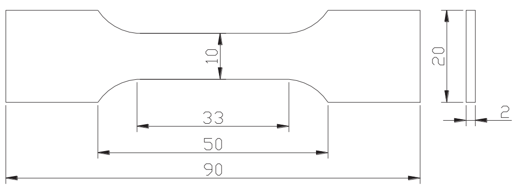
The pure resin used for the experiment was a mixture of 100 parts, by mass, Epikote 828 (a diglycidyl ether of bisphenol-A - DGEBA) (supplied by Robnor Resins, UK), 90 parts HY906 which is a curing agent type 1-methyl-5-norbornene-2,3-dicarboxylic anhydride (NMA) (supplied by Robnor Resins, UK) and 1 part DY062, Benzyldimethylamine (BDMA) (supplied by Huntsman Advanced Materials Ltd., UK) which is used as the accelerator. In order to prepare a series of nanocomposites with 5-25 wt% nanosilica content, the Epikote 828 resin was mechanically mixed with Nanopox F400 nanosilica/DGEBA masterbatch in a heated oil bath of 80^{o}C for 2 h. The mixture was degassed in a vacuum oven at 80^{o}C to remove the entrapped air, which then was blended with the appropriate stoichiometric amounts of NMA hardener and BDMA accelerator (based on the amount of DGEBA and the masterbatch) for 15 min. The nanomodified resin was afterwards poured into release-coated silicon moulds (plate and dogbone shape) and degassed in the vacuum oven before curing to remove any air entrapped in the mixture. Finally, the resin system was pre-cured at 80^{o}C for 2 h, cured at 120 ^{o}C for 3 h and post-cured at 150 ^{o}C for 4 h with a ramp rate of 1^{o}C/min followed by cooling down to room temperature at 1^{o}C/min.

2.2. Transmission Electron Microscopy (TEM)

The degree of dispersion of the silica nanosphere particle in the epoxy matrix was investigated using a TEM. TEM samples with a thickness of 85 nm were prepared using a Leica UC2 Ultra-microtome machine at room temperature. After cutting, sections were collected on 200-mesh copper grids. The specimens were examined using a FEI Tecnai TEM at an accelerating voltage of 80 kV. The images were captured using a Gatan MS600CW high resolution digital camera and collected using Gatan digital micrograph software at three different magnifications, 22500x, 115000x and 225000x.

2.3. Tensile tests

The dogbone shape specimens with a gauge length/width/thickness of 33mm/10mm/2mm were adhered with 1.5 mm thick glass fibre reinforced polymer (GFRP) composite end-tabs. This is to prevent a premature failure at grips. The dimensions of the specimen are illustrated in Fig. 1a. At least five specimens were tested for each system. Prior to testing, the actual width and thickness of the coupon at the gauge length was measured at 3 different points using a digital electronic Vernier calliper. The cross sectional area of the specimens was calculated by multiplying the mean width by the mean thickness. A Hounsfield Universal Testing machine with wedge type grips, as shown in Fig. 1b, was used for the tensile testing at a crosshead speed of 1 mm/min. A 10 kN load cell and a 25 mm gauge length clip-on extensometer were used to record the applied load and elongation data. These data were logged to a computer for analysis. The tensile tests were conducted based on British standard BS EN ISO 527-1 and -2 :1996. The tensile properties such as elastic modulus, strength and failure strain were determined based on the standard.



(a)

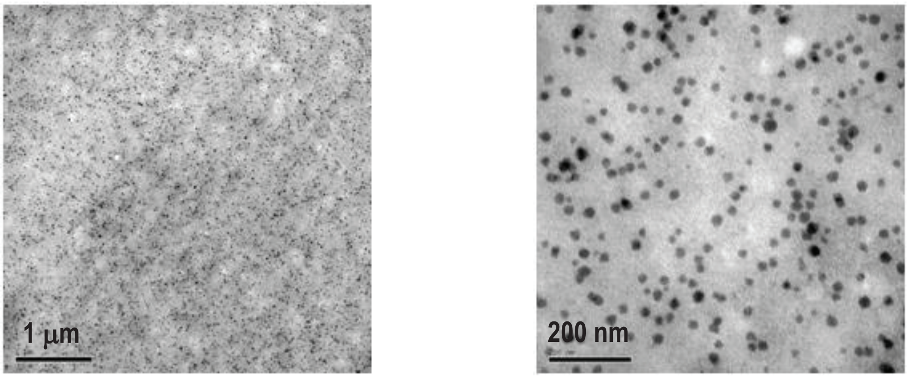
(b)

Fig. 1. Illustration of (a) dimensions of the dogbone shape specimen and (b) a Hounsfield universal testing machine with wedge type grips for tensile test.

3. Results and discussion

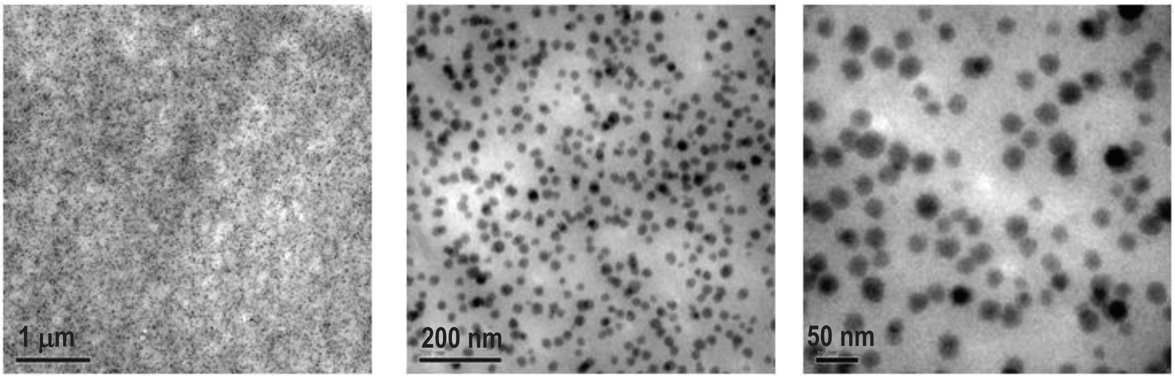
3.1. Morphology of nanosilica/epoxy nanocomposites

Homogeneous dispersion of nanofillers in a polymer is one of the major challenges in fabricating nanocomposites. Agglomeration of nanoparticles (usually in micrometer size clumps) often gives adverse effects on the thermal and mechanical properties of the epoxy. Hence, this does not represent the properties of a desired nanocomposite. In this study, a uniform distribution of nanosilica in Epikote 828 was achieved. This is supported by the TEM micrographs presented in Fig. 2. There was no agglomeration of the SiO_2 nanoparticles even at high volume fraction (see Fig. 2c). The spherical shape silica nanoparticles have a mean particle size of 20 nm and maximum diameter of 40 nm as observed at high magnification (see Fig. 2b(iii)). Since the TEM slice is approximately 85 nm thick these TEM images do not reflect the actual volume fraction of nanosilica in the matrix. The volume fraction of the nanosilica can be measured using thermo-gravimetry analysis (TGA).



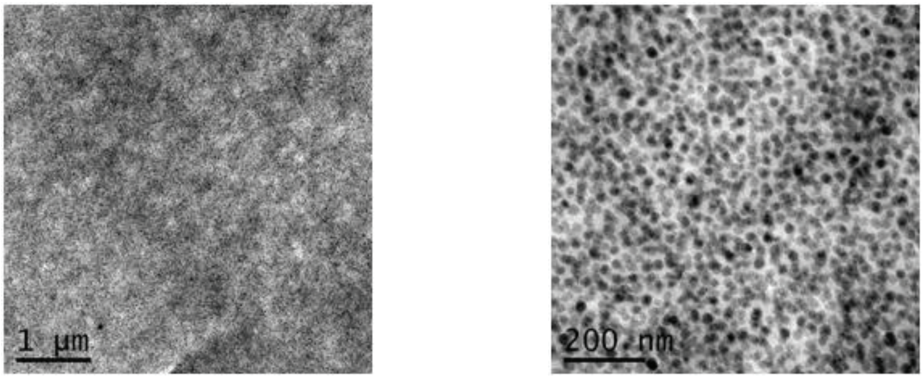
(i) 22500x (ii) 115000x

(a) 5 wt% nanosilica



(i) 22500x (ii) 115000x (iii) 225000x

(b) 13 wt% nanosilica



(i) 22500x (ii) 115000x

(c) 25 wt% nanosilica

Fig. 2. TEM micrographs showing a homogeneous dispersion of (a) 5 wt%, (b) 13 wt% and (c) 25 wt% silica nanospheres in Epikote 828 observed under three different magnifications. The spherical silica nanoparticles have a mean diameter of 20 nm and maximum diameter of 40 nm.

3.2. Tensile properties of pure polymer

Fig. 3 shows the stress-strain diagram of pure Epikote 828 polymer loaded in tension. All tested specimens were adhered with GFRP composite end-tabs to prevent failure at the grips as shown in Fig. 3(ii). Epoxy polymers are classified as brittle material because these materials usually fail in tension at relatively low values of strain. Fig. 3 shows that the Epikote 828 polymer fails with small elongation (of about 3% average failure strain) after the proportional limit (point A) is exceeded and the fracture stress (point B) is the same as the ultimate stress.

The diagram begins with a straight line where in this elastic region, the stress and strain are directly proportional. The tensile modulus of the material was measured from the slope of 0.1 to 0.25% tensile strain. Beyond point A, the deformation of the materials becomes permanent and the strain begins to increase more rapidly for each increment in stress. The stress-strain curve then has a smaller and smaller slope until it reaches point B then the material ruptures.

The tensile properties were determined based on Fig. 3 using equations given in British standard 527-1. The elastic modulus, tensile strength and failure strain of pure Epikote 828 polymer were 2.75 ± 0.02 GPa, 70.84 ± 1.08 MPa and 3.28 ± 0.08%, respectively.

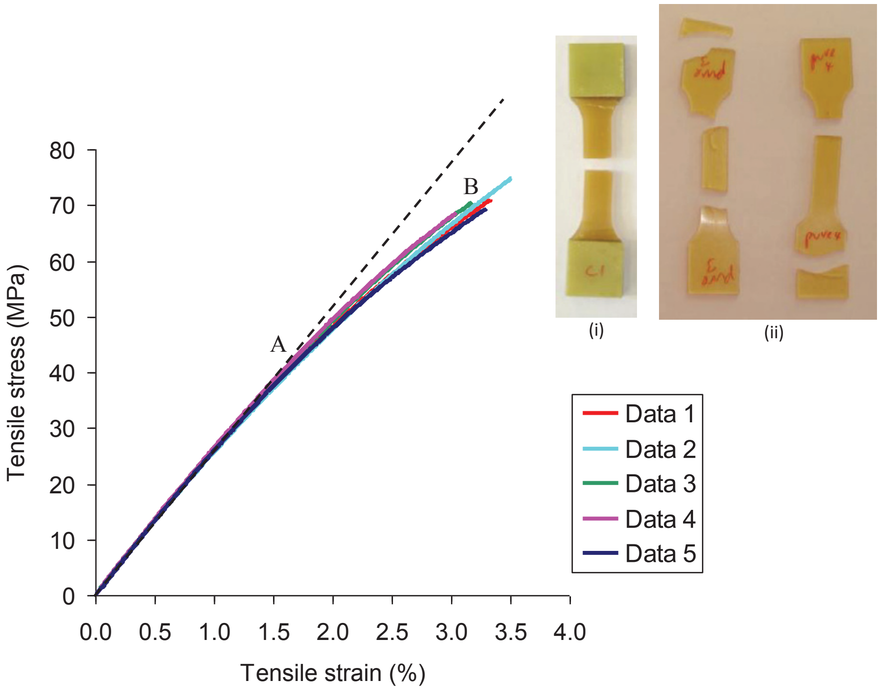


Fig. 3. Stress-strain curves of five dogbone shape specimens loaded in tension. All tested samples were adhered with GFRP composite end-tabs to prevent failure at grips. (i) and (ii) show examples of broken specimens; with tabs the specimen fails at gauge length and without tabs the specimen fails at grip.

3.3. Effect of nanosilica on tensile properties of Epikote 828 epoxy polymer

The effect of nanosilica on the tensile stress-strain response of the epoxy polymer is illustrated in Fig. 4. It can be seen that the presence of nanosilica enhanced the tensile stress-strain behaviour of the epoxy polymer. Nanocomposites exhibited higher tensile modulus (as measured at the initial slope of the graph) and strength without reducing its failure strain even at high nanosilica content. The increase in modulus is expected because the modulus of silica is about 70 Gpa [9]. In addition, the homogeneous dispersion of these high stiffness nanofillers in the matrix enhanced the fracture toughness of the system as indicated by the larger area under stress-strain curve of the nanocomposite system, see Fig. 4. As the tensile load increases, the matrix tries to elongate in its usual way. However, the nanofillers resist deformation. This results in smaller deformation compared to the neat polymer. Therefore, nanocomposites sustain more loads compared to the pure systems and contribute to a higher tensile modulus and strength.

Table 1 summarises the tensile properties of nanomodified system compared to the pure resin. It was found that the addition of nanosilica improved the tensile properties of the epoxy. For instance, the addition of 13 wt% nanosilica into the epoxy matrix enhances the tensile modulus by 21%, tensile strength by 20% and failure strain by 10%. The highest content of nanosilica in the epoxy (25 wt%) give a remarkable increase in tensile modulus and strength of about 38% and 24%, respectively, compared to the neat polymer without sacrificing the strain to failure. This suggests that the nanofiller-matrix interaction is very strong therefore the nanocomposites exhibited higher strength compared to the pristine polymer.

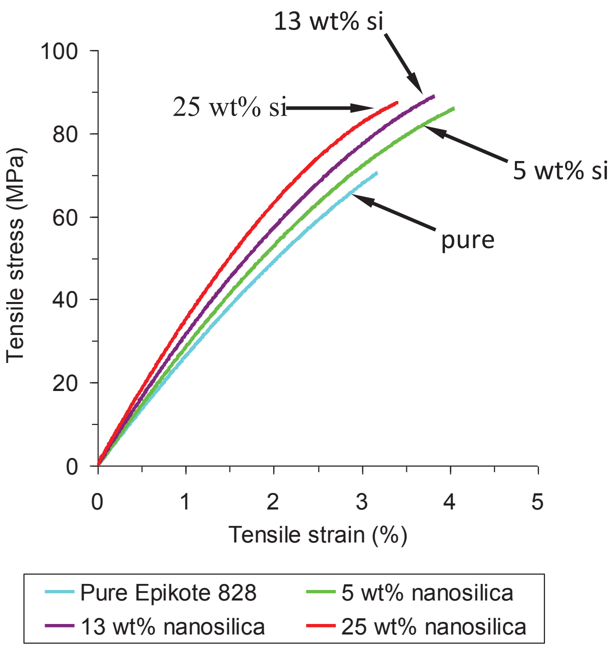


Fig. 4. Typical tensile stress-strain curves of nanosilica-modified Epikote 828 compared to the neat epoxy.

Table 1. Tensile properties of the unmodified and nanomodified Epikote 828 resin with various concentrations of silica nanoparticles.

Material properties	Pure resin	Nanomodified system (NMS)		
		5 wt% si	13 wt% si	25 wt% si
Tensile modulus, E_t (GPa)	2.75 ± 0.02	3.08 ± 0.04	3.33 ± 0.04	3.80 ± 0.04
Tensile strength, $\sigma_{u,t}$ (MPa)	70.84 ± 1.08	79.42 ± 4.00	85.25 ± 2.47	88.11 ± 1.41
Tensile strain at break, $\varepsilon_{f,t}$ (%)	3.28 ± 0.09	3.47 ± 0.30	3.62 ± 0.23	3.52 ± 0.22

4. Conclusion

A series of nanocomposites was developed based on nanosilica and Epikote 828 epoxy resin. TEM micrographs revealed that well-dispersed and non-agglomerated nanocomposite systems were produced. The performance of the nanocomposites was evaluated via static uniaxial tensile tests. Nanocomposites offer higher tensile stiffness and strength when compared to the neat polymer without sacrificing the failure strain of the material. In addition, the tensile stress-strain response showed that the presence of nanosilica improves ductility and toughness (based on a larger area under graph). The nanomodified resin is a promising candidate for developing high damage resistant and tolerant composite structures.

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